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MOLD AND CRUCIBLE COATINGS

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U.S. Army Research and Development Command

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This report details the investigation of coating materials and their application to the graphite crucible and molds used in melting and casting DU-0.75 Ti alloy.
Different types of washes were investigated: zirconium oxide, zirconium silicate, yttrium oxide, titanium carbide (used as an undercoat), and boron nitride. The parameters investigated in the application of these coatings were number of burnouts of the crucible, method of cleaning,

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surface condition of the crucible, temperature of the crucible during painting, number and time between coats, and method of drying the crucible.

The method of applying the wash affects its adherence to the graphite. The more thorough the drying of the first coat, the more effectively the coating protects the crucible. Drying in still air was detrimental to the success of most washes. Both oven or vacuum drying of the coated crucibles improved the adherence of most washes.

Performance of yttrium oxide washes was dependent on the physical properties of the CMC binders. Yttria coating of the molds improved ingot surface quality, compared with zirconia coated or uncoated molds.

Generally, zirconium oxide washes outperformed titanium carbide undercoated systems and yttrium oxide washes in the elevated temperature tests and in chemistry and ultrasonic results. Keywords:)

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INVESTIGATION OF MOLD AND CRUCIBLE COATINGS AND THEIR APPLICATION CONTRACT DAAK10-84-C-0056

INTRODUCTION

PURPOSE

The purpose of this MM&T was to evaluate various coating materials for application to the graphite components - crucible, pour cup, and ingot molds-used in the melting and casting of depleted uranium-3/4 titanium alloy for large caliber penetrators.

The coating material used on the graphite components, commonly called a "wash", is intended to prevent attack by the molten metal. If the wash fails either by chemical attack or loss of adhesion, the melt will become contaminated with carbon. In DU-3/4 Ti melts, if carbon exceeds the solubility limit, it forms titanium carbide (TiC) inclusions. These inclusions have a negative impact upon chemistry and ultrasonic test results. Since they are lower density than molten DU-3/4 Ti, the TiC particles tend to float toward the top of the ingot during solidification. Significant carbon contamination can lead to Ti and C levels in excess of the specified limits. In addition, experience at Nuclear Metals, Inc. (NMI) has shown inclusion stringers are a significant cause of ultrasonic rejects.

The Army sponsored this MM&T to identify coatings with improved performance, ones that would increase chemistry and utltrasonic yields.

BACKGROUND

In late 1983, NMI initiated a company funded R&D program to investigate crucible washes. At that time, results of the initial chemical analyses were unacceptably low - falling between between 84 and 91%. (Yield is calculated by dividing the number of chemically acceptable ingots divided by the total number of ingots tested.) Both Ti and C were causes for rejection. Although retests improved the yields, it was a very unsatisfactory situation due to the lost material and increased labor hours and testing costs. In addition, retests had a negative impact on a smooth flow of material within the plant.

A technical evaluation of the low chemistry yields indicated the crucible wash as a very significant contributor to the problem. At the time, NMI obtained zirconia wash from Didier-Taylor Refractories. The wash consisted of a dry powder mixture of zirconium oxide, sodium silicate, and a proprietary plasticizer. Upon mixing with water, the powder yielded suspensions with highly variable physical properties. NMI contacted several vendors (Didier-Taylor, Remet Ceramics, and ZYP Coatings) and a DU facility (Rocky Flats) about their wash products. Trips were made to these facilities to observe their production and quality control procedures. Technical discussions were held with these vendors as to improved formulations which could be evaluated by NMI.

NMI developed several test procedures during the IRAD to evaluate the different coatings - viscosity, settling rate, and elevated temperature firing tests. (These same procedures were employed in this MM&T and are fully explained later in the report.) Different application techniques and methods of crucible preparation were also evaluated in conjunction with the firing tests. The variables investigated were number of crucible burnouts, number of crucible uses, cleaning method, number of coats, time between coats, preheating of the crucible prior to coating, drying method, and wash formulations.

The IRAD results are incorporated into the MM&T results in order to provide a complete body of technology for the DU industry.

The NMI IRAD led to the selection of Remet zirconia wash #2 for production melts. In addition, an improved application procedure was implemented. Chemistry yields improved dramatically, 97 to 99% on an ingot basis. Production melts made with Remet #2 zirconia served as the baseline comparison for the MM&T trials.

The Army funded this MM&T with the expectations of finding a cheaper or more effective wash. Task A involved the investigation of the various coating materials. The washes were subjected to numerous evaluations - viscosity, settling rate, and reactivity tests. In Task B, the methods of application were evaluated and an optimum procedure developed. Crucible firing tests determined the effectiveness of the wash adherence to the graphite. Task C tested two coating systems selected from among the washes evaluated in Tasks A and B. Six full scale melts were cast, and ingot chemistries were evaluated. In Task D, five melts made with the best wash selected from Task C were processed through ultrasonic evaluation in order to verify that the wash was not harmful to chemical and ultrasonic results.

TASKS A AND B

WASH SELECTION AND APPLICATION

BACKGROUND

Selecting an optimal application procedure for the washes(Task B) was done concurrently with the evaluation of the wash systems (Task A). Subscale melts, to determine the

reactivity of the wash with molten uranium, could not be run until the best method of application was determined by the elevated temperature crucible firing tests. Therefore, Tasks A and B were run simultaneously. The NMI IRAD program had already demonstrated that all wash systems survive the firing tests if the crucibles are new. Therefore, all MM&T firing tests were run with used crucibles. The variables investigated in Task B of the MM&T were number of crucible burnouts, cleaning method, time between coats, preheating of the crucible prior to coating, drying method, and wash formulations.

The MM&T scope of work for Task A was originally set up to include four (4) sub-scale melts in which the reactivity of two (2) wash systems with uranium could be explored. After completing the original scope of work, new wash systems were discovered which merited evaluation. Task A was then expanded to include additional wash evaluations and three (3) additional subscale melts. However, by this stage, an optimal application procedure had already been established in Task B. Therefore, the new washes were all evaluated using the established procedures.

PROCEDURES

WASH PREPARATION AND TESTS

A list of the washes evaluated in both the MM&T and IRAD efforts is in Table 2. Each of the washes was mixed to the formulation recommended by the vendor. ZYP washes (zirconium oxide "Z" and "ZO", yttrium oxide, titanium carbide, and boron nitride) were pre-mixed, ready for use after re-mixing. Remet and Didier supplied dry zirconium oxide powder mixtures containing sodium silicate binder and a proprietary plasticizer. These washes were prepared by adding a weight ratio of four (4)

parts powder to one (1) part agitated water followed by mixing for a period of one hour. Foseco's Terrapaint 550, a zirconium silicate paste, also required a 4 to 1 mix ratio with water.

Rocky Flats supplied NMI with a one gallon sample of their mixed yttria wash. This wash is basically a 45% formulation made with BASF Wyandotte sodium carboxymethylcellulose (CMC); the Wyandotte CMC is no longer marketed. At NMI, all the 45% formulation yttria washes were made by adding a dry mixture of 1000 grams of yttrium oxide and 30 grams of sodium CMC to 1200 milliliters of water. The mixture was then ball milled for at least four hours. There were differences in the physical characteristics of the CMC's viscosities used to make batches of yttria wash; CMC is an organic binder resembling water glass. The preparation of the 35% and 50% yttria formulations was considerably different - instead of mixing all the ingredients at one time, the CMC was thoroughly dispersed in the water before any yttria addition. In both cases, 22.5 grams of CMC was added to 750 milliliters of water; this mixture was ball milled for at least 72 hours. Then 404 or 750 grams of yttrium oxide were added to make 35% or 50% formulations, respectively. The slurries were ball milled at least 24 hours before crucible application. (The theory behind this wash system is that an undercoat of the thinner 35% wash seals the graphite pores better than a coat of the 50% wash - the top coat.)

Two tests which classify the physical properties of the wash are viscosity and settling rate. The ultimate goal at NMI is to machine spray a uniform coat of wash on the bottom and sides of the crucible. Machine sprayed coatings are more uniform in thickness, more reproducible, and much better in limiting the radiation exposures to the operators. If a wash is

too thin, coverage of the graphite surface may be insufficient; whereas, if the wash is too thick, it may not be capable of being sprayed, so viscosity also plays an important part.

Viscosity is measured with a Zahn No. 4 Viscosimeter (see Figure 1). A representative sample of the mixed slurry is taken; the Zahn cup is pushed through the mixture in order to fill the volume inside the cup. The Zahn cup is then lifted quickly and as it breaks the surface of the slurry, a timer is started. The Zahn value is the time (in seconds) necessary to drain the cup of the wash mixture; the acceptable range is 26 to 38 seconds.

Settling rate should be slow because thorough resuspension is difficult in these washes due to their fine particle size distribution. A 100 milliliter sample (see Figure 2) of the thoroughly mixed slurry is observed for the amount of settling which occurs with time. The maximum acceptable separation is five (5) ml. of discernable, clear liquid within a 24 hour period.

ELEVATED TEMPERATURE ADHESION

In order to test the adhesion of the coating to the graphite, crucibles were first hand painted with vertical stripes of various coatings (see Figure 3). The empty crucibles were then placed into a furnace used for large caliber penetrator production melts. Under a vacuum of less than 200 microns, the temperature was raised to 1400°C for ten minutes using the heating cycle given in Table 1. The crucibles were then cooled under vacuum. The fired crucibles were then visually inspected, and the coatings were qualitatively rated for adhesion to the graphite.

Results of the NMI IRAD program demonstrated that all washes, independent of method of application, survived the firing tests in new, pre-conditioned crucibles. Figure 4 illustrates the typical success of washes painted inside a new crucible. The only firing test failures during the IRAD work occurred in used crucibles; therefore, the MM&T experiments were restricted to used crucibles (see Tables 4).

The different parameters evaluated in the painting of the crucibles (during this MM&T and the NMI IRAD) were number of crucible burnouts, crucible usage, cleaning preparation, preheating prior to coating, number of coats, time between coats, drying method, and wash formulations. Burnout is a means of facilitating skull removal in which used crucibles are heated in air so that the uranium metal converts into oxide ashes.

The variables investigated under cleaning preparation during the IRAD included dry rags for a new crucible, rotary brushed or rotary brushed and wiped with an alchohol dipped rag for used crubibles. Usually a very fine dust film can be felt on a graphite surface. The theory behind using the rags (wet or dry) was to remove this dust in case coating adherence was diminished by this thin layer. IRAD results showed no detectable differerce in adherence, so this variable was dropped from the MM&T tests.

Preheating the crucible prior to coating should help the coating thickness to be uniform and changes the adhesion characteristics (depending on the number of coats). The number of coats evaluated in the IRAD program were either one or two

coats. Most washes performed worse in firing tests with a single coat as opposed to two coats, all other factors being equal. Therefore, the MM&T tests were all made with two coats.

Time between coats was either 20 or 60 minutes in the IRAD program. This parameter did not appear to have much effect on elevated temperature adhesion. Most of the MM&T runs were either 20 or 30 minutes between coats. Three drying methods were evaluated - drying for 4 hours in a 350°F oven, applying a vacuum hose to the inside of the painted crucible for at least two hours, and drying the painted crucible uncovered, overnight in a hood. The MM&T confirmed that hood drying was very detrimental to coating adherence.

REACTIVITY

The ultimate test for a wash is its reactivity with molten uranium. Subscale melts (200 to 300 kg. of charge instead of the normal 800 kg.) were made in Task A with used crucibles hand painted according to the application procedure established in Task B with four of the most effective wash systems. The application procedure (developed in Task B) consisted of one crucible burnout, cleaning, preheating to about 110°F, and coating. Thirty minutes after applying the first coat, the second coat was applied. Drying was achieved by inserting a vacuum hose into the crucible for a minimum of two hours. The cycle used for the melts is shown in Table 1; six (or seven) holes were plugged with graphite so that a standard NMI nine (9) hole pour cup could be employed to cast the smaller charge of virgin DU (of known carbon content) and titanium sponge. The ingots from lots N25 to 28 and N33 to 35 were then sampled in the top, middle and bottom, and analyzed for titanium, carbon, iron, copper, nickel, silicon, and the coating constituent.

RESULTS AND DISCUSSION OF RESULTS

PHYSICAL PROPERTIES

Zirconium oxide and silicate washes are a beige color and dry into a hard, sandy coating. Yttrium oxide washes are white and dry into a thin, powdery coating. The titanium carbide wash is black and dries into a hard, smooth coating. The boron nitride wash is white and dries into a hard, smooth coating. It appeared that yttria, being composed of a finer particle size distribution than zirconia, packs into a wet sand-like consistency and is harder to resuspend.

The results of the wash property evaluations from both the NMI IRAD and this MM&T are listed in Table 3. Pre-mixed Didier #2 ZrO_2, Remet #1 ZrO_2 (with a finer particle size distribution) and Remet #3 ZrO_2 (with increased plasticizer) were so thick that they could not be applied evenly or easily. Dry Didier #1 ZrO_2 and Y_2O_3 made with Carbose DHT CMC settled out quickly, making resuspension very difficult to control. For these reasons, the above mentioned washes were not evaluated in the firing tests of this MM&T. ZYP Y_2O_3 and yttria with Carbose DM-LVT CMC had borderline settling rates. Didier #3 and ZYP "ZO" zirconia washes closely resembled the Remet #2 in formulation and in physical properties. Table 18 includes these reasons as a cause for elimination from the remainder of the work.

ELEVATED TEMPERATURE ADHESION

Five washes were ruled out as candidates for the firing tests based on previous NMI IRAD experience; these washes were yttria made with Hercules 7M CMC, Didier's #1 and #2 zirconia washes, and Remet's #1 (finer particle size distribution) and #3

(increased plasticizer) zirconia washes. Based on the wash properties and previous firing test results, the coatings selected for the first ten crucible firing tests (see Table 4) in Task A were as follows:

Wash	Number Tests
Reduced Remet #2 ZrO_2	10
Foseco $ZrSiO_4$	10
Hercules 7M1 Y_2O_3	10
Carbose DM-LVT Y_2O_3	10
ZYP ZrO_2 "Z"	5
Hercules 6CTL1 Y_2O_3	5
ZYP BN	5
ZYP Y_2O_3	5

Figures 5 to 8 contain photographs of the crucibles after firing to 1400°C. The area under evaluation is the lower half of the crucible where the molten metal pool would be in contact with the coating. The two washes used as references for comparison in the firing tests were Remet #2 zirconia, currently being used in standard production, and Rocky Flat's yttria formulation.

The crucible shown in Figure 5 was dried in a 350°F oven for four hours after having had two coats of wash applied 30 minutes apart. The Remet ZrO_2 wash is entirely intact; on the other hand, chunks of yttria wash made with the Hercules 6CTL1 CMC are missing. The details in the photograph in Figure 6 are harder to discern, but the stripe painted with ZYP "Z" ZrO_2 wash was entirely missing and flakes were hanging off the stripes of yttria washes made with 6CTL1 and DM-LVT CMC. The crucible in Figure 6 was preheated, 30 minutes between coats, and dried with a vacuum hose for two hours. Both crucibles in Figures 7 and 8 were dried in the hood after hand painting. In

Figure 7, the crucible was preheated to 110°F and allowed 20 minutes between its two coats. The crucible in Figure 8 went through two burn-out cycles and was dried 20 minutes between coats. In both photographs, the ZYP "Z" ZrO_2 wash demonstrated the poorest adherence.

Table 4 summarizes the results of the initial Task A crucible firing experiments. Performance ratings were qualitative, depending on the nature and location of the peeled coating. No failure (0) meant that the coating survived the test; other designations in the table are "F" if coating fell off completely, "RS" if coating was ready to fall off (crumbling of the wash upon slight touch), "TF" if the top coat fell off, but the undercoat remained, "FL" if the failure occurred only at the top liquid line, "RSL" if the coating was very loose and crumbly at the liquid line only, "S" if the wash fell off only in small areas, and "SL" if these patches of bare graphite occurred only at the liquid line. The "liquid line" refers to the top line where the molten metal extends; i.e., the height of the molten metal pool. Realistically, the wash must survive in order that the molten uranium will not be exposed to bare graphite.

The best performance was demonstrated by Remet #2 ZrO_2 which is the wash currently used at NMI for production melts. The poorest coating was ZYP's "Z" ZrO_2 wash; the entire coating layer fell off, completely exposing the graphite. Poor ratings were given to three yttria washes - ZYP pre-mixed, Hercules 6CTL1, and Carbose DM-LVT CMC yttria systems. Thermodynamic calculations for the boron nitride wash revealed poor stability with uranium. Overall, zirconia washes outperformed the yttria and boron nitride washes in the adhesion tests. Table 18 lists these results as a cause for elimination from further consideration.

From Table 4, it is obvious that drying of the coated crucibles in the hood created failures with most methods of preparation and with most washes. The two drying procedures which appeared to work the most satisfactorily in the firing tests were:

1. Single burn out, no preheat, 30 minutes between coats, four (4) hours drying in a 350°F oven

2. Single burn out, 110°F preheat, 30 minutes between coats, 2 hours drying with a vacuum hose

These two procedures were repeated in the last two firing tests to assure reproducibility. Vacuum drying (Procedure 2) was the most practical procedure easily adaptable to full-scale production work. Since either method gave good results, vacuum drying, method #2 above, was chosen for the remainder of the melts. The heat, whether before or after coating, is required to drive off the moisture which might vaporize later, leaving a fizzure in the wash coating.

Generally, it appeared that the more thorough the drying of the first coat, the more effectively the coating protected the graphite. If the first coat was still wet when the second coat was applied, then if failure did occur, it usually resulted in both coats of wash falling off. On the other hand, if the first coat was dry before application of the second coat, then if failure did occur, it was usually displayed with only the top coat falling off while the first coat still remained intact.

The first two washes selected for the reactivity portion of the Task A subscale melts were Foseco's $ZrSiO_4$ and Y_2O_3 made with Hercules 7M1 CMC. Remet #2 ZrO_2 was not used for any of the subscale melts because its behavior in contact with molten uranium was already being established with the production runs.

REACTIVITY

Reactivity between the selected wash formulation and molten DU-3/4 Ti was investigated in subscale melts (198 kg. charges of derby and Ti sponge) which were melted and poured. The intent of the original scope of Task A was to make four melts, each with three 20.5-inch castings, to be chemically analyzed. However, due to the small thermal mass and low head height,a substantial amount of the subscale melt froze in the pour cup. As a result, the first melt, N25 made with Foseco zirconia, yielded three ingots 13 to 15 inches in length. In subsequent melts, seven of the nine holes in the pour cup were plugged. This approach gave improved yields of cast metal. Melts N27 and N28 made with yttria plus 7M1 CMC yielded two full length ingots. However, melt N26 with Foseco zirconia, made in a different furnace, again experienced rapid freezing in the pour cup and yielded two short (about 15 inch long) ingots.

All of the ingots were sampled in the top, middle, and bottom in areas corresponding to standard sampling locations for extruded rods (except the middle which is normally not tested), and analyzed for titanium and carbon content. In addition, one ingot in each melt was analyzed for the trace elements (Fe, Cu, Ni, Si) and for the coating constituent (zirconium in the first two melts and yttrium in the last two melts). As can be seen in the results found in Table 5, all of the melt chemistries were well within M833 specification. The trace element results show that the coatings did not react with the molten metal. Although the melts made with Foseco's $ZrSiO_4$ are slightly higher in silicon than the other melts, the Si level is still half the maximum allowable limit.

Some porosity was visible in the saw cut faces of the billets (located about 3.75" from the top of the ingot) in two subscale melts - N25 (Foseco $ZrSiO_4$) and N28 (Y_2O_3 with 7M1

CMC). The short billets from lot N25 had very tiny voids not exceeding a width of 1/8" (see Figure 9). The maximum diameter of the voids exhibited in the full length billets from lot N28 were about 1/4" (see Figures 9 and 10). This porosity is not believed to be affected by the different crucible washes, but rather, it is due to the solidification pattern occurring in the mold.

Foseco $ZrSiO_4$ paste could have numerous complications. Mixing and handling on a full scale basis would be inconsistent since transfer into the mixing container would demand more labor, and its heaviness may create water losses due to splashing during addition. Also, NLO has reported that this paste has a limited shelf life because of bacterial growth and that it presents a storage problem during periods of cold temperatures due to its freezing tendency. NLO personnel also felt that this paste could contribute a 20 ppm pickup in silicon content. These reasons are reflected in Table 18.

EXPANDED TASK A (Wash Selection) WORK

The chemistry results from the subscale melts were not a dramatic improvement over the current chemistry results of production melts made with Remet #2 zirconium oxide wash. Through continued discussions with the vendors during Task A, new wash systems were discovered which merited evaluation. Rocky Flats supplied NMI with a gallon of pre-mixed yttrium oxide used in their production melts. This sample acted as a reference for all the other yttria washes evaluated. The engineer who formulated the Remet washes moved over the Didier-Taylor firm. Didier-Taylor offered an improved dry zirconium oxide powder for NMI to evaluate. ZYP claimed to have an improved (different plasticizer) zirconium oxide (pre-mixed) wash which was thixotropic and never settled out. Also, ZYP believed that titanium carbide wash could effectively reduce the diffusion of carbon from the graphite crucible if it is used as an undercoat in the

crucible. Another yttria wash system was recommended on the theory that a 35% first coat of yttria could help seal the pores of the crucible surface and make a smoother contact surface for the second coat (50% yttria). As a result, NMI and G. Cadigan of the Army agreed that evaluations of these added washes should be made so the Army PCO provided extra funds for the added evaluations.

Eleven washes were evaluated in four additional crucible firing tests:

Wash	Test Run Number
Remet #2 ZrO_2	All
"Rocky Flats" Y_2O_3*	1-2
Didier #3 ZrO_2*	3-4
ZYP "ZO" ZrO_2*	1-2
7M1 - 45% Y_2O_3	1-4
7M1 - 35% + 50% Y_2O_3*	1-4
TiC* + "ZO" ZrO_2*	1-2
TiC* + 7M1 - 45% Y_2O_3	3-4
TiC* + 7M1 - 50% Y_2O_3*	2-3
TiC* + Remet #2 ZrO_2	2-3
TiC* + Didier #3 ZrO_2*	3-4

* New washes

The best method of wash application (established in Task B of this program) was used to paint these four crucibles. Used crucibles were burned out, cleaned with a rotary brush, pre-heated to 110°F, painted, and dried. The second coat was applied after a waiting period of 30 minutes (with a vacuum hose inserted in the crucible). A vacuum hose was inserted into the crucible for at least two hours to dry the two wash coats.

Table 4 summarizes the results of these firing tests. These washes had a much higher success rate than the washes fired in the ten previous firing tests. None of the three washes which had mild failures (Rocky Flats Y_2O_3, TiC plus 7M1 Y_2O_3, and Remet #2 ZrO_2), failed consistently.

Didier #3 and ZYP's "ZO" zirconia are basically made up of the same constituents as Remet #2 zirconia. The 35%/50% yttria wash combination was felt to be too labor intensive for for production and to be similar to the 45% yttria wash previously tested for reactivity with uranium. Therefore, these three washes were not selected for the reactivity portion of Task A, as listed in Table 18.

Therefore, two TiC combination washes were chosen for the reactivity test. In order to avoid possible doubts as to the cause of failure being due to the top or bottom coat, it was decided to use proven washes for the top coats - Remet #2 ZrO_2 and Hercules 7M1-45% Y_2O_3.

In order to cast three full length ingots, the amount of derby and titanium charged for the three subscale melts in the expanded part of the program was increased to 290 kg. Lot N33 was melted in a crucible painted first with titanium carbide, then with Remet #2 zirconium oxide. The crucibles used in lots N34 and N35 also had a first coat of titanium carbide, but their second coats were Hercules 7M1-45% yttrium oxide. The same melting procedure as that used in the previous subscale melts in Task A was employed.

All of the ingots were sampled in the top, middle, and bottom locations, and tested for titanium and carbon content. In addition, one ingot in each melt was tested for the trace elements (Fe, Cu, Ni, Si) and for the coating constituent

(zirconium in the first melt and yttrium in the last two melts). Titanium and carbon are standard analyses for every ingot. As can be seen in the results found in Table 5, all of the melt chemistries were within M833 specifications.

NMI has developed a method of rating melt chemistries based on their titanium and carbon analyses. The rating system has a scale of 0 to 10 (10 being the best). Each rating value has a special classification as explained in Table 6. Failures create processing delays and material losses ranging from one blank to one billet to several billets to the entire lot. This subjective scale takes the failures into account and rates the melt lower as the number of failures increase.

Listed in Table 7 are the ratings for the seven subscale melts in Task A. According to this rating system, the washes tested for reactivity with uranium would be ranked as follows:

Best	TiC + Remet #2 ZrO_2
	Foseco $ZrSiO_4$
	Hercules 7M1-45% Y_2O_3
Worst	TiC + 7M1-45% Y_2O_3

CONCLUSIONS

TASK A - WASH SELECTION

1. Two new wash systems merited study in the Task C production melts. These washes were:

   a - 45% yttria made with Hercules 7M1 CMC binder.

   b - titanium carbide undercoating with a Remet #2 zirconia top coat.

2. Remet #2 zirconia, the current NMI production crucible wash, could be eliminated from consideration for Task C because there was ample data on its behavior.

3. Didier #3 and ZYP "ZO" zirconia washes could also be eliminated for Task C evaluation because of their close resemblance to Remet #2 zirconia.

4. NLO's reported Si pickup, bacterial contamination, and freezing observations, as well as the paste's handling and mixing complications precluded any further work with Foseco's zirconium silicate.

5. Boron nitride is thermodynamically unstable in contact with molten uranium, and hence unsuitable as a wash material.

6. There is no advantage to the combined use of 35% and 50% Y_2O_3 washes made with Hercules 7M1 CMC - their mixing and application is labor intensive. In addition, the combination closely resembles the 45% Y_2O_3 formulation.

7. Performance of yttria wash systems was dependent on the physical properties of the CMC binders.

TASK B - WASH APPLICATION

1. For production purposes, a single crucible burnout, a 110°F preheat, 30 minutes between coats, and 2 hours drying with a vacuum hose represents the most practical application procedure.

2. A single burnout, no preheat, 30 minutes between coats, and 4 hours drying in a 350°F oven also is a satisfactory method. However, this procedure would require additional capital equipment for production.

TASK C - FULL SCALE MELTS

PROCEDURE

Full scale (800 kg.) melts (N74 to 79) of virgin derby plus titanium sponge were made in hand painted, used crucibles as required in the scope of work. The melt cycle was the standard cycle used for M833 production lots. The two wash systems investigated were (1) yttrium oxide made with Hercules 7M1 CMC and (2) ZYP's titanium carbide in conjunction with Remet #2 zirconium oxide. The application procedure (developed in Task B) consisted of burning out the crucible, cleaning, preheating to 110°F, and coating. Thirty minutes after applying the first coat, the second coat was applied. Drying was achieved by inserting a vacuum hose for a minimum of two hours. Metallographic and chemistry samples were removed from the ingots and evaluated. The sampling plan for the metallography samples is listed in Table 8. The chemistry samples were removed from the top and bottom of each ingot in areas corresponding to the sampling locations for the extruded rods.

Melt lots N74-76-78 had crucibles painted with two coats of 45% Y_2O_3 made with Hercules 7M1 CMC; the pour cups were also painted with the yttria wash. Melt lots N75-77-79 had a first coat of ZYP's TiC, then a top coat of Remet #2 ZrO_2 applied to the crucible surface. The pour cups were painted with the zirconate wash. Melts N74 to 77 were cast into molds with uncoated walls (per NMI's production procedures). Melts N78 and 79 had three types of molds: three with bare walls, three with yttria coated walls, and three with zirconia coated walls.

RESULTS AND DISCUSSION OF RESULTS

NMI has found that the zirconium oxide wash used for the crucibles does not act as a good releasing agent. Solidified metal tends to attach itself to the graphite surface more strongly than desired. The outcome is difficulty in removing the metal and also rough surfaces. As a result, M833 melts use magnesium zirconate washes in the pour cups and mold bottoms, but the mold walls are left uncoated.

The chemical analyses of the ingots are found in Table 9. Chemistries did not show any effects from the mold coating. At this point, improved ingot surface is not beneficial because surface imperfections are not deep enough to show up in the final machined cores. If the extrusion diameters were to be reduced, yttria would become important.

Metallographic evaluation of the ingots in the longitudinal direction did not reveal any unusual inclusions, such as the wash constituent. It appeared that the TiC plus ZrO_2 melts were slightly cleaner than the Y_2O_3 melts. Finely (up to 20 μ) dispersed titanium carbide precipitates were visible throughout the matrix. Occasional agglomerates containing uranium oxide and titanium carbide particles were also identified. Typical photo-micrographs from these ingots can be found in Figures 11 to 13.

Listed in Table 10 are the chemistry ratings for the six full scale melts of Task C. The wash systems were equal with regards to carbon ratings, but TiC plus Remet ZrO_2 was slightly better than Y_2O_3 in titanium ratings.

A summary of the Ti and C ratings for all of the yttria coated melts made at NMI under IRAD's, previous contracts and this MM&T, is presented in Table 11. Melts made with yttria

wash historically at NMI appear to have a higher incidence of titanium segregation and/or failures than normally found with zirconia wash. In Subtask B of Contract DAAK10-81-C-0105, yttria melts using Hercules 7M CMC were processed through ultrasonic testing. Of the four melts evaluated, one melt had unacceptable rod chemistry (titanium segregation and carbon in excess of 80 ppm), and two melts had high ultrasonic reject levels. Samples showing ultrasonic indications were sectioned and metallographically evaluated to reveal high density areas of stringered inclusions, not porosity.

CONCLUSIONS

1. A coat of ZYP's titanium carbide overlayed with a coat of Remet #2 zirconium oxide was the most promising wash system for Task D.

2. Mold coatings of yttria or zirconia did not produce any significant variation in chemistry.

3. Yttria coating of the molds improves ingot surface quality, compared with zirconia coated or uncoated molds.

TASK D - PRODUCTION PROVE-OUT

PROCEDURES

In Task D, charges of 800 kg were made consisting of one derby plus DU-3/4 Ti class A recycle and titanium sponge alloying addition per the NMI M833 procedure. The used crucibles were hand coated with the combination titanium carbide plus zirconium oxide wash system per the established procedure. (Machine coating was not employed because the coater can only handle one type of wash, unless modifications and additions are

made to the existing equipment.) Melt lots M15 through 19, cast in Task D, had a combination of TiC plus Remet ZrO_2 washes applied to their crucibles. The pour cups were coated with zirconate wash, and the mold walls were left bare. All five melts (M15 to 19) were extruded in copper, solution heat treated in vacuum for two hours at 850°C, quenched at 16 ipm into agitated water, rotary straightened, aged for 16 hours at 360°C, and pre-machined. The extruded bars were analyzed for chemical composition, and the pre-machined blanks were ultrasonically tested.

RESULTS AND DISCUSSION OF RESULTS

The chemistry results for the extruded rods are listed Table 12. All ingots met chemistry specifications, and no titanium segregation was incurred. The carbon pick-up ranged from 13 to 21 ppm - normal in production melts. The zirconium content was as high as 55 ppm, so some pickup was experienced when compared to the two reference melts which ranged 19 to 25 ppm Zr. (However, these two production melts are really not enough to use as a baseline comparison so this pickup may not be significant.) Apparently, the wash system protected the graphite during most of the melt.

Another method of determining reactivity of the wash with uranium is the lack of inclusions in the metal. Pre-machined blanks were ultrasonically tested on the NMI manual unit with the results listed in Table 13. Two of the five lots had rather high rejection rates - M16 and M19. Thirty-nine production M833 lots which were melted with two coats of Remet zirconia wash and which were melt and cast during the same time period, averaged a 2.3% rejection rate. In comparison, these five MM&T melts averaged a 3.5% rate and would have to be rated as mediocre to poor.

Based on an evaluation of the ultrasonic traces, only 1/3 of the rejects were associated with centerline porosity. Most of the reject indications (see Table 14) were attributable to inclusions. It is possible that some of the inclusions may be detached wash constituent particles, in addition to the typical oxides and carbides formed by metal attack with the graphite components. (EDAX analyses would be required to identify constituent particles.)

A qualitative, judgemental rating system has been employed at NMI to compare pulse echo traces. The rating ranges from 0 (for low background with a limited number of spikes) to 4 (for high background with a substantial number of spikes). Each pulse echo trace was reviewed and rated; the results are listed in Table 15. Table 16 has a summary of the rating averages by billet and lot. Typically, as the bar location moves from front to rear of the extruded rod, the pulse echo traces tend to worsen probably due to higher inclusion content at the top of the ingot. This pattern held true in most cases. Most billets within the lot were equally rated - no billet stands out as being much better than any other. This uniformity among the castings suggests that the inclusions did not form in the mold (due to dirty walls, for example), but formed either in the crucible or in the pour cup. Time in the pour cup is very short, so it is very unlikely that the metal reacts with the pour cup wall in that short a time span. Therefore, it appears most likely that these inclusions are being formed in the crucible.

CONCLUSIONS

1. The titanium carbide - zirconium oxide wash system produces chemistries typical of a standard M833 production melt.

2. The titanium carbide - zirconium oxide wash system is detrimental to yields at ultrasonic testing. Inclusions formed during melting accounted for two-thirds of the reject indications.

TASK E - SAFETY AND ECONOMIC ANALYSIS

SAFETY AND ECONOMIC ANALYSIS

A comparison of the dual wash system vs. the current production method was performed as it impacted on safety concerns and the cost of equipment, material, and labor.

Since ZYP's titanium carbide wash is already pre-mixed, the safety concerns for eye and skin contact, ingestion, and inhalation would be less than currently encountered with the Remet zirconia wash which is a dry powder initially. The mixture contains water and an organic binder, and it is stable and non-flammable. The protective gear for the skin and eyes currently used in mixing the zirconia wash is more than adequate for handling the titanium carbide.

Although ultrasonic yields on this new material was 1.2% lower than production during the same melting period, no yield effect was considered in this analysis. The conditions used in this economic analysis are as follows:

a. Annual basis of 500 melts per year.

b. A catalog price of $250/gal. for TiC and $5/lb. for Remet ZrO_2. However, ZYP offers a 15% discount for an annual purchase of 50 gallons of TiC.

c. Capital cost of equipment and facilities estimated at $18,135.

d. No change in labor hours.

e. Operating cost increase due to the change calculated to be $5,280 in the first year and inflated by 4% per year thereafter.

f. Depreciation expense calculated under ACRS guidelines: 5 years for equipment.

g. Cash flows projected on an after tax basis using an effective tax rate of 46%.

Results of the economic analyses show the wash system of TiC + ZrO_2 would be more expensive (see Table 17) than the application of two coats of ZrO_2 used in current production. The raw material is more expensive, and new hardware would be required to convert the crucible cleaning station to spray two different coats of wash (see Figure 14). Associated with the new equipment are initial, installation and maintenance costs. The total initial outlay for the equipment would be $18,135, and the added cost in raw material would be $5,280/year.

The conclusion of this analysis is that there is no economic benefit to be gained in changing from the current Remet crucible coating to a dual TiC and Remet coating. It is estimated there would be a negative cash flow of $23,422 at the end of five years. Based on this expense and the facts that chemistry results were equal and ultrasonic results were worse, NMI does not recommend switching to the new wash system in melting and casting of DU-3/4 Ti alloy.

CONCLUSIONS

1. There are added costs associated with the titanium carbide - zirconium oxide wash system. These costs include converting the coating station to handle two different washes, increased maintenance and operation times, and increased raw material costs. It is estimated there would be a negative cash flow of $23,422 over a period of five years.

2. Implementation of the titanium carbide - zirconium oxide wash system in production is not warranted. The absence of a significant improvement in metal quality does not justify the added costs.

CONCLUSIONS AND RECOMMENDATIONS

Table 18 summarizes the performance of each of the washes and the reason each was removed from further consideration. Major conclusions to be drawn from this MM&T are:

1. Zirconium oxide washes outperformed the other wash system.

2. The standard crucible preparation adopted for production was reconfirmed as being an effective means of applying wash - burning out once, preheating to 110°F, applying the first coat, waiting 30 minutes before applying the second coat, and drying 2 hours with a vacuum hose.

3. An alternative, satisfactory method of wash application is burning out once, applying a first coat, waiting 30 minutes before applying a second coat, and drying four hours in a 350°F oven.

4. Yttrium oxide as a mold wash improves ingot surface quality.

5. Mold wash is not detrimental to chemistry results.

6. Even though the titanium carbide plus zirconium oxide wash system yielded material which met M833 chemistry

specifications, ultrasonic reject rates were slightly higher (by 1.2%) than standard production melts made with Remet #2 zirconium oxide wash.

7. Since metal quality was not significantly improved by the TiC plus ZrO_2 wash system and this wash system is more costly, implementation of this wash in production is not warranted.

Table 1.

Test Cycle

Firing Test	Subscale Melts
50 KW for 10 minutes - typically 362°C	50 KW for 10 minutes - typically 280°C
80 KW for 35 minutes - typically 1400°C	80 KW for 35 minutes - typically 1220°C
60 KW until the temperature reaches 1400°C	60 KW until all metal is molten - typically 70 minutes
Hold at 1400°C for 10 minutes, adjusting power as necessary so as not to exceed 1410°C.	Hold for 25 minutes, adjusting power setting as necessary so as not to exceed 1410°C. Target temperature is 1400°C.
Typical total cycle time was 55 minutes.	Typical total cycle time was 105 minutes.

Table 2

Washes Investigated

Name	Form	Mixing Ratio[a]	Binder
		Zirconium Oxide ZrO_2	
[b]Didier #1	Powder	4:1	Na_2SiO_4 + Plasticizer
[b]Didier #2	Pre-mixed	-	Na_2SiO_4 + Plasticizer
[b]Didier #3	Powder	4:1	Na_2SiO_4 + Plasticizer
[b]Remet #1	Powder	4:1	Na_2SiO_4 + Plasticizer
[b]Remet #2	Powder	4:1	Na_2SiO_4 + Plasticizer
[b]Remet #3	Powder	4:1	Na_2SiO_4 + Plasticizer
[b]ZYP "Z"	Pre-mixed	-	Unknown
ZYP "ZO"	Pre-mixed	-	Unknown
		Zirconium Silicate $ZrSiO_4$	
[b]Foseco	Paste	4:1	Unknown
		Yttrium Oxide Y_2O_3	
"Rocky Flats"	Powder	0.9:1	BASF Wyandotte CMC
[b]45%	Powder	0.9:1	Hercules 7M CMC
[b]45%	Powder	0.9:1	Hercules 7M1 CMC
35%	Powder	0.6:1	Hercules 7M1 CMC
50%	Powder	1.0:1	Hercules 7M1 CMC
[b]45%	Powder	0.9:1	Hercules 6CTL1 CMC
45%	Powder	0.9:1	Carbose DHT CMC
[b]45%	Powder	0.9:1	Carbose DM-LVT CMC
[b]ZYP "Y"	Pre-mixed	-	Unknown
		Titanium Carbide TiC	
ZYP	Pre-mixed	-	Unknown
		Boron Nitride BN	
[b]ZYP	Pre-mixed	-	Unknown

[a] Parts by weight, powder to water
[b] Investigated in NMI IRAD

Table 3. Wash properties

Wash description	Zahn Value[a] sec.	Settling[b] (ml) at time (hours) <7	24	48
Zirconia:				
Foseco Paste	17	-	0	0
Remet Fine #1	Thick[c]	0	0	0
Remet Reduced #2	27/36	0	0	0
Remet #3	Thick[c]	0	0	0
ZYP Wet "Z"	20	0	0	1
ZYP Wet "ZO"	Thick[c]	0	0	0
Didier Dry #1	14/26	2	12	29
Didier Wet #2	Thick[c]	0	0	0
Didier Dry #3	38	0	0	0
Yttria:				
Rocky Flats Mixed	70[d]	0	0	0
Hercules 7M-45%	Thick[c]	1	2	2
Hercules 7M1-45%	13/23	1	4	4
Hercules 7M1-35%	17	-	4	4
Hercules 7M1-50%	40	-	0	1
Hercules 6CTL1-45%	29	0	2	3
Carbose DHT-45%	11	-	21	21
Carbose DM-LVT-45%	20	-	5	7
ZYP Wet	16	1	6	8
ZYP TiC	15	0	0	0
ZYP BN	36	0	0	0

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a Viscosity is measured with Zahn No. 4 Viscosimeter.

b Settling is the amount of liquid which has separated in 100 milliliter graduated cylinder.

c Thick refers to the viscosity being so great that the cup plugged.

d Measured 41 seconds on Zahn No. 5 Cup. Estimated conversion to Zahn No. 4 Cup is 70 seconds.

TABLE 4

Original Task A
Firing Experiments

TEST	BURNOUT		CLEANING			SURFACE	BEFORE COATS	TIME BETWEEN COATS				DRYING AFTER COATS		
	Reg.[a]	Dbl.[b]	Mch.[c]	Vac.[d]	Alc[e] Wipe	Very Pitted	110°F Pre-heat	1/3 hour	1/2 hour	1 hour	Heat Gun	350°F Oven	Vac.[d] Hose	Nite Hood
1	X		X			X		X				X		
2		X	X	X		X				X				X
3		X	X		X	X		X			X			X
4	X		X			X	X		X				X	
5	X		X			X	X	X						X
6	X		X			X	X		X				X	
7	X		X			X	X	X					X	
8	X		X			X		X			X		X	
9	X		X		X	X			X			X		
10	X		X			X	X	X				X		

a - Regular burnout refers to one single burnout and cleaning.
b - Double burnout refers to burning out, cleaning, then burning out and cleaning again.
c - Machine cleaning involves a mechanical rotary brush cleaning.
d - Vacuum involves applying vacuum suction to the inside of the crucible.
e - Alcohol wipe refers to wiping the ID with a rag dipped in alcohol.

TABLE 4 (Cont'd.)

FIRING TEST EXPERIMENTS
FOR EXPANDED TASK A WORK

CODE LETTER	EXPT. NO. 1	2	3	4
R	X	X	X	X
RF	X	X		
D			X	X
Z	X	X		
SY	X			X
LY	X			X
TZ	X	X		
TS			X	X
TY		X	X	
TR		X	X	
TD			X	X

Code: R= 2 coats Remet #2 zirconia

RF= 2 coats Rocky Flats yttria

D= 2 coats Didier #3 zirconia

Z= 2 coats ZYP's "ZO" zirconia

SY= 2 coats 45% yttria made with Hercules 7M1 CMC

LY= 1 coat 35% Y_2O_3 (7M1 CMC), then 1 coat 50% Y_2O_3 (7M1 CMC)

TZ= 1 coat ZYP's TiC, then 1 coat ZYP's "ZO" Zirconia

TS= 1 coat ZYP's TiC, then 1 coat 45% Y_2O_3 (7M1 CMC)

TY= 1 coat ZYP's TiC, the 1 coat 50% Y_2O_3 (7M1 CMC)

TR= 1 coat ZYP's TiC, then 1 coat Remet #2 ZrO_2

TD= 1 coat ZYP's TiC, then 1 coat Didier #3 ZrO_2

Table 4 (Cont'd.) Crucible Firing Test Results for IRAD and Part I of Task A

Special Preparation	Time Between Coats	ZIRCONIA				YTTRIA					BN
		Remet 1	Remet 2	ZYP 2	Foseco	ZYP	7M	DM-LVT	7M1-45	6CTL1	ZYP
Used crucible - dried 4 hours at 350°F after coating											
	60[d]	0	-	0	-	-	RS	-	-	-	-
	30	-	0	F	0	-	-	S	S	S	-
	30	-	0	-	TF[b]	TF/RSL	-	TF/RSL	TF/FL	-	TF
Pre-heat	20	-	0	-	S/FL	SL/TF	-	TF/FL	TF/S	-	TF/S
Used crucible - dried 2 hours with vacuum hose after coating											
	60[d]	0	-	0	-	-	TF	-	-	-	-
Pre-heat	30	-	0	F	0	-	-	S	0	TF	-
Pre-heat	30	-	0	-	0	0	-	TF	0	-	0[b]
	20[c]	0	-	0	-	-	0	-	-	-	-
	20 + Vac	-	0	-	S	S	-	S	S	-	S
Pre-heat	20	-	0	-	TF	S	-	0[b]	TF	-	TF
Used crucible - dried overnight in hood after coating											
	60[d]	-	-	F	-	-	F	-	-	-	-
	60[d]	-	0	FB	-	F	-	-	-	-	-
	20[d]	-	0	F	-	F	-	-	-	-	-
	20[d]	-	F	FL	-	F	-	-	-	-	-
	20[d]	-	F	F	-	-	-	-	-	-	-
	20[d]	-	F	F	-	-	-	-	-	-	-
	20[d]	F	-	F	-	-	F	-	-	-	-
Pre-heat	20	-	0	F	S	-	-	RS	F	TF	-
Double BO[c]	20 + Vac	-	U	F	RS	-	-	S	S	S	-
Double BO	60	-	F	F	0	-	-	S	0	S	-

a - 0 = no failure
F = fell off completely
RS = ready to spall off
TF = top coat fell off
FL = coating fell off at liquid line
RSL = ready to spall off at liquid line
S = some patches where coating fell off completely
SL = some patches at liquid line where coating fell off completely

b - These ratings were difficult to determine - best approximations are given, but uncertain.

c - Double BO stands for burning out the crucible twice with a cleaning operation after each cycle.

d - IRAD test result.

Table 4 (Cont'd.). Crucible Firing Test[a] Results for Part II of Task A

	ZIRCONIA			YTTRIA			TITANIUM CARBIDE PLUS[c]				
Run No.	Remet #2	ZYP ZO	Didier #3	Rocky Flats	7M1 45%	7M1 35/50[b]	ZYP ZO	7M1 45%	7M1 50%	Remet #2	Didier #3
1	0	0	-	0	0	0	0	-	-	-	-
2	FRS	0	-	RSL	-	-	0	-	0	0	-
3	0	-	0	-	-	-	-	RSL	0	0	0
4	0	-	0	-	0	0	-	0	-	-	0

a All crucibles tested were used crucibles preheated to 110°F and dried two (2) hours with a vacuum hose after painting. Thirty (30) minutes were allowed between the application of the two coats; a vacuum hose was inserted into the crucible during this waiting period.

b First coat was the 35% yttria formulation; second coat was the 50% yttria formulation.

c First coat was ZYP's TiC; second coat varied - ZYP's ZO, 45% Y_2O_3, 50% Y_2O_3, Remet #2, Didier #3.

d 0 = no failure
FRS = few bubbles ready to spall off
RSL = few bubbles at liquid line ready to spall off.

Table 5

Results[a] of Chemical Analysis of Subscale Melts

ID	Location	Ti	C	Fe	Ni	Cu	Si	Zr	Y
Derby	2295 T	-	.007	-	-	-	-	-	-
N25[b]	1 T	.71	.003	39	8	3	78	14	-
	1 M	.70	.003						
	1 B	.72	.004						
	2 T	.72	.003						
	2 M	.70	.004						
	2 B	.69	.004						
	3 T	.71	.004						
	3 M	.70	.004						
	3 B	.70	.004						
Derby	2295 B	-	.006	-	-	-	-	-	-
N26[b]	1 T	.70	.004						
	1 B	.70	.003	34	6	3	79	30	-
	2 T	.71	.004						
	2 B	.70	.003						
Derby	2295 M	-	.006	-	-	-	-	-	<10
27[c]	1 T	.73	.004	33	4	1	23	-	10
	1 M	.70	.005						
	1 B	.70	.004	34	5	1	31	-	10
	2 T	.72	.005						
	2 M	.71	.005						
	2 B	.72	.005						
Derby	2311 M	-	.005	-	-	-	-	<3	-
N28[c]	1 T	.73	.006						
	1 M	.74	.005						
	1 B	.70	.004						
	2 T	.71	.005	8	5	1	21	-	<10
	2 M	.72	.005						
	2 B	.70	.005	10	6	2	20	-	42
M833	spec.	69-.79	<.008	50 MAX	50 MAX	50 MAX	150 MAX	-	-

a Titanium and carbon are reported in weight percent; all other elements are in parts per million.

b. Lots N25 and N26 were made in crucibles painted with Foseco's $ZrSiO_4$. Lot N25 made three short ingots; whereas, lot N26 made two short ingots.

c. Lots N27 and N28 were made in crucibles painted with 7M1 Y_2O_3. Both lots produced two full length ingots.

Table 5 (Cont'd.)

Results[a] of Chemical Analysis of Subscale Melts

ID	Location	Ti	C	Fe	Ni	Cu	Si	Zr	Y
Derby N33[b]	3138 T	-	.0025	-	-	-	-	-	-
	1 T	.73	.004						
	1 M	.74	.004						
	1 B	.72	.003						
	2 T	.75	.006						
	2 M	.74	.008						
	2 B	.73	.003						
	3 T	.73	.004	10	4	3	51	12	-
	3 M	.72	.003						
	3 B	.72	.003	11	4	3	49	15	-
Derby N34[c]	3138 B	-	.0025	-	-	-	-	-	-
	1 T	.76	.002						
	1 M	.72	.004						
	1 B	.72	.003						
	2 T	.72	.004	12	5	3	20	-	30
	2 M	.72	.003						
	2 B	.72	.003	11	4	3	20	-	41
	3 T	.72	.004						
	3 M	.74	.005						
	3 B	.74	.004						
Derby N35[c]	3150 T	-	.003	-	-	-	-	-	-
	1 T	.73	.005						
	1 M	.73	.008						
	1 B	.72	.004						
	2 T	.77	.007						
	2 M	.74	.006						
	2 B	.72	.004						
	3 T	.75	.007	8	12	5	24	-	21
	3 M	.72	.006						
	3 B	.72	.005	9	12	6	24	-	43

a. Titanium and carbon are reported in weight percent; all other elements are in parts per million.

b. Lot N33 was made in crucible painted first with titanium carbide, then with Remet #2 zirconia.

c. Lots N34 and N35 were made in crucibles painted first with titanium carbide, then with 7M1-45% yttria formulation.

Table 6. Melt Chemistry Rating System

Rating	No. of Failures	Comments
		Titanium
10	0	Average Ti 0.725 to 0.744 -no spread.
9	0	Other average below and above the "10" spec. - no spread.
8	0	Average spread $\geq$ 0.02% independent of average Ti.
7	1	
6	2	
5	3	
4	4	
3	5	
2	6	
1	7	
0	8	
		Carbon
10	0	Rear average C up to 49 ppm.
9	0	Rear average C $\geq$ 50 ppm.
8	1	
7	2	
6	3	
5	4	
4	5	
3	6	
2	7	
1	8	
0	9	

Table 7. Chemistry Ratings of the Subscale Melts

Lot	Ti[a]	C[b]	Wash System
N25	9	10	Foseco $ZrSiO_4$
N26	9	10	Foseco $ZrSiO_4$
N27	9	9	Hercules 7M1-45% Y_2O_3
N28	8	9	Hercules 7M1-45% Y_2O_3
N33	10	10	TiC + Remet #2 ZrO_2
N34	8	10	TiC + 7M1-45% Y_2O_3
N35	8	9	TiC + 7M1-45% Y_2O_3

[a] Standard production melts are normally sampled in the front and rear of the extruded rod. For this MM&T, the ingot Ti chemistries were rated using the corresponding locations - bottom and top.

[b] Standard production melts are normally sampled in the front and rear of the extruded rod. For this MM&T, the ingot C chemistries were rated using all three values - bottom, middle, and top.

Table 8

Microstructure Sampling Plan

For Task C

Melt Number	Comment	No. Ingots	Location Top	Location Bottom	Total Samples
-	2 Derbies	-	x		2
1	Ctg. 1	1	x^3	x	4
1	Ctg. 1	1	x	x	2
3	Ctg. 1	1	x^3	x	4
3	Ctg. 1	1	x	x	2
5	Ctg. 1 molds (3)	4	x	x	8
2	Ctg. 2	1	x^3	x	4
2	Ctg. 2	1	x	x	2
4	Ctg. 2	1	x^3	x	4
4	Ctg. 2	1	x	x	2
6	Ctg. 2 molds (3)	4	x	x	8
A	Std. production	1	x^3	x	4
B	Std. production	1	x	x	2

* All micros will be taken from the center of the cross-section, except melts 1 to 4 in Task C - the tops of the ingots (one per melt) will be sampled in the edge, mid-radius, and center of the cross-section.

Table 9
Task C Chemistries
Two Coats of Yttria

ID	Locn.	Ti	C	Fe	Ni	Cu	Si	Zr	Y
Derby	3288 T		.009						
	B		.006	.0071	average			--	24
	3276 T		.006					2	22
N74	1 T	.72	*.014	42	5	2	12		
	B	.71	.004	25	6	2	12		
	2 T	.73	.004					--	34
	B	.72	.004					--	46
	3 T	.73	.007						
	B	.72	.004						
	4 T	.71	.004						
	B	.72	.004						
	5 T	.75	.005						
	B	.72	.004						
	6 T	.73	.006						
	B	.71	.005						
	7 T	.74	.005						
	B	.72	.004						
	8 T	.75	.004						
	B	.72	.004						
	9 T	.75	.007						
	B	.72	.004						
Rating		9→8	8						
Ave.		.724	.0052						
Derby	3278 T		.003					--	<10
	B		.004	.0044	average				<10
	3276 M		.007					2	22
N76	1 T	.72	.004						
	B	.74	.005						
	2 T	.75	.005						
	B	.74	.006						
	3 T	.75	.007						
	B	.74	.007						
	4 T	.75	.005						
	B	.74	.004						
	5 T	.76	.005						
	B	.74	.004						
	6 T	.76	.006	10	6	1	16		
	B	.73	.005	11	7	1	16		
	7 T	.75	.006					--	47
	B	.75	.005					--	44
	8 T	.74	.006						
	B	.75	.006						
	9 T	.75	.007						
	B	.74	.006						
Rating		10	9						
Ave.		.744	.0055						

* Exceeded 80 ppm specification.

Table 9 (Cont'd.)
Task C Chemistries
Two Coats of Yttria

ID	Locn.	Ti	C	Fe	Ni	Cu	Si	Zr	Y
Derby	3283 T		.006					--	<10
	B		.003	.0039	average				
	3267 M		.002						
N78	1a T	.75	.003					--	39
	B	.74	.005					--	30
	2a T	.74	.005	13	5	1	17		
	B	.73	.003	13	4	1	14		
	3a T	.74	.004						
	B	.73	.003						
	4b T	.75	.003					--	24
	B	.74	.004					--	29
	5b T	.75	.004					--	35
	B	.74	.003					--	49
	6b T	.72	.004						
	B	.72	.004						
	7c T	.74	.005						
	B	.73	.006						
	8c T	.74	.005					1	--
	B	.73	.003					1	--
	9c T	.73	.007					1	--
	B	.72	.003					1	--
Rating		10	10						
Ave.		.736	.0041						
Rating									

a= Molds were uncoated.
b= Molds were coated with yttria wash.
c= Molds were coated with Remet Zirconia wash.

Table 9 (Cont'd.)
Task C Chemistries
$TiC + ZrO_2$

ID	Locn.		Ti	C	Fe	Ni	Cu	Si	Zr	Y
Derby	3274	T		.006						
		B		.008	.0079	average				
	3276	B		.110					2	22
N75	1	T	.74	.006						
		B	.73	.005						
	2	T	.73	.005						
		B	.74	.008						
	3	T	.75	.008						
		B	.73	.004						
	4	T	.73	.004						
		B	.73	.006						
	5	T	.74	.005	12	8	2	38		
		B	.73	.006	12	9	1	41		
	6	T	.76	.006					26	--
		B	.73	.006					24	--
	7	T	.75	.007						
		B	.73	.006						
	8	T	.74	.006						
		B	.74	.005						
	9	T	.74	.005						
		B	.74	.006						
Rating			10	9						
Ave.			.738	.0058						
Derby	3284	T		.004						
		B		.003	.0031	average				
	3267	T		.002						
N77	1	T	.74	.002						
		B	.74	.002						
	2	T	.75	.001						
		B	.74	.002						
	3	T	.76	.002						
		B	.74	.002						
	4	T	.73	.001						
		B	.73	.003						
	5	T	.75	.002						
		B	.75	.003						
	6	T	.74	.002						
		B	.74	.002						
	7	T	.76	.004	17	5	2	46		
		B	.73	.002	15	6	2	45		
	8	T	.75	.002					11	--
		B	.74	.002					11	--
	9	T	.76	.002						
		B	.73	.002						
Rating			10	10						
Ave.			.743	.0021						

Table 9 (Cont'd.)
Task C Chemistries
Ti C + ZrO_2

ID	Locn.	Ti	C	Fe	Ni	Cu	Si	Zr	Y
Derby	3281 T		.002						
	B		.005	.0039	average			1	--
	3267 B		.005						
N79	1c T	.79	.003					41	--
	B	.73	.003					55	--
	2c T	.73	.004					52	--
	B	.73	.008					43	--
	3a T	.75	.006	14	11	2	33		
	B	.72	.002	15	12	3	34		
	4b T	.74	.003					--	40
	B	.72	.007					--	35
	5a T	.74	.004					37	--
	B	.72	d.027					33	--
	6a T	.74	.003						
	B	.74	.003						
	7b T	.74	.004						
	B	.73	.002						
	8b T	.73	.003					--	41
	B	.73	.002					--	44
	9c T	.72	.004						
	B	.72	.005						
Rating		10	8						
Ave.		.734	.0052						

Two Standard Production Melts
Two Coats of ZrO_2 (for Reference)

ID	Locn.	Ti	C	Fe	Ni	Cu	Si	Zr	Y
Derby	3523 T		.005						
945	2 T							20	
	B							19	
Rating		10	10						
Ave.		.739	.0032						
Recycle	?		<.008						
938	4 T							25	
	B							24	
Rating		9	10						
Ave.		.703	.0030						

a= Molds were uncoated.
b= Molds were coated with yttria wash.
c= Molds were coated with Remet zirconia wash.
d= Exceeded 80 ppm specification.

Table 10

Chemistry Ratings of the
Task C Melts

Lot	Ti[a]	C[a]	Wash System
N74	8.5	8	Hercules 7M1-45% Y_2O_3
N76	10	9	Hercules 7M1-45% Y_2O_3
N78	10	10	Hercules 7M1-45% Y_2O_3[b]
N75	10	9	TiC + Remet #2 ZrO_2
N77	10	10	TiC + Remet #2 ZrO_2
N79	10	8	TiC + Remet #2 ZrO_2[b]

a. Standard production melts are normally sampled in the front and rear of the extruded rod. For this MM&T, the ingot Ti and C chemistries were rated using the corresponding locations - bottom and top.

b. Mold wash varied in these melts.

Table 11

Chemistry Ratings of All
NMI Y_2O_3 Melts

Melt	Ti	C	Date
N5	0	0	
N8	10	10	June, 83
N9	9	10	
N11	8	10	
N27	9	9	July, 84
N28	8	9	
319	9	9	
23	0	7	
28	9	9	Aug., 84
36	10	10	
62	9	10	
521	9	9	
24	0	0	
25	10	10	
26	9	10	Oct., 84
27	9	10	
29	10	10	
35	9	9	
N34 (above 1 coat of TiC)	8	10	Dec., 84
N35 (above 1 coat of TiC)	8	9	
N74	9→8	8	
N76	10	9	Feb., 85
N78	10	10	

Table 12
Task D Chemistries

JD	Locn.	Ti	C	Fe	Ni	Cu	Si	Zr
Derby	3266	--	.0058					
Recycle	N75-8	.74	.0055	ave.	.0016			
	N75-9	.74	.0055					
M15	1 F	.73	.003					
	R	.73	.003					
	2 F	.73	.003					
	R	.73	.003					
	3 F	.73	.002					
	R	.73	.003					
	4 F	.73	.003					
	R	.73	.004					
	5 F	.73	.003	14	7	2	38	26
	R	.73	.004	14	6	2	39	25
	6 F	.73	.002					
	R	.73	.003					
	7 F	.73	.003					
	R	.78	.003					
	8 F	.73	.003					
	R	.73	.003					
	9 F	.73	.003					
	R	.73	.002					
Rating		10	10					
Ave.		.733	.0029					
Derby	3716	--	.0021					
Recycle	N75-1	.735	.0055	ave.	.0028			
	N75-2	.735	.0065					
M16	1 F	.73	.004					
	R	.74	.004					
	2 F	.74	.004					
	R	.74	.005					
	3 F	.74	.005					
	R	.75	.005					
	4 F	.74	.004					
	R	.73	.005					
	5 F	.73	.001					
	R	.77	.004					
	6 F	.74	.004	13	15	5	35	26
	R	.74	.005	14	15	5	32	21
	7 F	.74	.004					
	R	.74	.005					
	8 F	.74	.004					
	R	.74	.004					
	9 F	.74	.005					
	R	.74	.003					
Rating		10	10					
Ave.		.741	.0042					

Table 12 (Cont'd.)
Task D Chemistries

ID	Locn.		Ti	C	Fe	Ni	Cu	Si	Zr
Derby	3675		--	.0027					
Recycle	N75-3		.74	.006	ave.	.0033			
	N75-4		.73	.005					
M17	1	F	.73	.004					
		R	.73	.006					
	2	F	.75	.004					
		R	.74	.004					
	3	F	.74	.005					
		R	.75	.004					
	4	F	.75	.006					
		R	.75	.005					
	5	F	.75	.005					
		R	.75	.004					
	6	F	.75	.006					
		R	.75	.005					
	7	F	.75	.004	11	9	3	42	58
		R	.75	.007	11	9	3	40	65
	8	F	.75	.004					
		R	.74	.007					
	9	F	.74	.007					
		R	.75	.004					
Rating			9	9					
Ave.			.746	.0051					
Derby	3727		--	.0024					
Recycle	N75-5		.735	.0055	ave.	.0030			
	N75-6		.745	.006					
M18	1	F	.75	.006					
		R	.75	.004					
	2	F	.75	.004					
		R	.75	.005					
	3	F	.75	.005					
		R	.75	.005					
	4	F	.75	.004					
		R	.76	.004					
	5	F	.76	.005					
		R	.75	.004					
	6	F	.75	.004					
		R	.76	.005					
	7	F	.75	.004					
		R	.76	.005					
	8	F	.75	.004	13	9	3	37	27
		R	.75	.004	14	10	3	32	28
	9	F	.75	.005					
		R	.76	.005					
Rating			9	10					
Ave.			.753	.0046					

Table 12 (Cont'd.)
Task D Chemistries

ID	Locn.	Ti	C	Fe	Ni	Cu	Si	Zr
Derby	3268	--	.0031					
Recycle	N75-7	.74	.0065	ave.	.0037			
	N74-9	.735	.0055					
M19	1 F	.74	.005					
	R	.75	.006					
	2 F	.73	.005					
	R	.76	.007					
	3 F	.74	.006					
	R	.74	.008					
	4 F	.73	.005					
	R	.74	.006					
	5 F	.73	.006					
	R	.74	.007					
	6 F	.74	.006					
	R	.74	.004					
	7 F	.73	.006					
	R	.74	.005					
	8 F	.74	.005					
	R	.74	.005					
	9 F	.74	.005	12	7	2	35	51
	R	.74	.005	11	7	2	35	51
Rating		10	9					
Ave.		.740	.0058					

Table 13

Ultrasonic Test Results

Lot	Inspected	Rejected	% Rejected
M15	111	3	2.7
M16	111	6	5.4
M17	111	1	0.9
M18	100	3	3.0
M19	110	6	5.5
All 5	543	19	3.5

Table 14

Type of Ultrasonic Rejects

Lot - Bar	Channel*	Defect(s)
M15-5-3	PE	Inclusion
7-13	PE	One of several inclusions
8-10	PE	Inclusion
M16-3-13	PE	Pipe and its inclusions
5-6	PE	One of many inclusions
5-10	PE	One of many inclusions
6-10	PE	Inclusion
7-6	PE	One of a few inclusions
7-11	PE	Two of many inclusions
M17-8-13	PE	Pipe and its inclusions
M18-4-13	PE	Pipe and its inclusions
5-13	PE	Pipe and its inclusions
6-13	PE	Pipe and its inclusions
M19-2-11	PE	Inclusion
2-13	PE	Pipe and its inclusions
9-4	PE	Inclusion
9-8	PE	One of a few inclusions
1-4	RS	Inclusion
9-12	RS	One of a few inclusions

* PE stands for the pulse echo channel.
RS stands for the right shear channel.

TABLE 15

Qualitative Rating[a] of the Pulse Echo Charts for Lot M15

ID	Rating	ID	Rating	ID	Rating
1- 1	0	4-1	0	7-1	0
2	1	2	1.5	2	1
3	1	3	1	3	1
4	1.5	4	1	4	1.5
5	2	5	2	5	1.5
6	1	6	1.5	6	2
7	1.5	7	2	7	2.5
8	1.5	8	2	8	1.5
9	1.5	9	2	9	1.5
10	2	10	2	10	2
11	1.5	11	2	11	2
12	2	12	2	12	2
13	---	13	1.5	13	4.5[b]
2- 1	0	5- 1	0	8- 1	0
2	0.5	2	0.5	2	0
3	1	3	1	3	1
4	1	3	1	4	1.5
5	1	5	1	5	1.5
6	1.5	6	1	6	2
7	1	7	0.5	7	2.5
8	1	8	1	8	2
9	1.5	9	1.5	9	2
10	2	10	1.5	10	2.5[b]
11	2	11	1.5	11	2.5
12	2	12	1.5	12	2.5
13	---	13	2.5[b]	13	---
3- 1	0.5	6- 1	0	9- 1	0
2	0.5	2	0	2	0
3	1	3	0.5	3	1
4	1	4	1	4	1
5	1	5	1	5	1.5
6	2	6	1.5	6	1
7	2	7	1	7	2
8	1.5	8	1	8	1.5
9	2.5	9	1.5	9	1
10	2.5	10	2	10	1
11	2	11	1.5	11	1.5
12	2	12	2	12	1
13	---	13	---	13	---

a. Scale: 0 = Low background with few spikes to 4 = high background with many spikes.

b. Reject Bar

TABLE 15 (Cont'd.)

Qualitative Rating[a] of the Pulse Echo Charts for Lot M16

ID	Rating	ID	Rating	ID	Rating
1- 1	0	4-1	0	7-1	0
2	2	2	0.5	2	0.5
3	1	3	0.5	3	1
4	1	4	1	4	1
5	1.5	5	1	5	1.5
6	1	6	1	6	2[b]
7	1.5	7	1.5	7	2
8	1.5	8	2	8	2.5
9	2	9	1.5	9	2.5
10	1.5	10	2	10	2.5
11	2.5	11	2	11	3[b]
12	2.5	12	3	12	3
13	2	13	---	13	---
2- 1	0	5- 1	0.5	8- 1	0
2	1	2	0.5	2	0.5
3	0.5	3	1.5	3	0.5
4	1	3	1.5	4	0
5	1.5	5	1	5	0
6	1.5	6	2.5[b]	6	1
7	2.5	7	1.5	7	2
8	2	8	1.5	8	1.5
9	2.5	9	2	9	2
10	2	10	3.5[b]	10	2
11	2.5	11	3.5	11	1.5
12	2.5	12	---	12	2.5
13	2.5	13	---	13	---
3- 1	1	6- 1	1	9- 1	0
2	1	2	0.5	2	0
3	1.5	3	1.5	3	1
4	1	4	1.5	4	1
5	1.5	5	1	5	2
6	1.5	6	2	6	2
7	2	7	2	7	2
8	1.5	8	2	8	2
9	2.5	9	2	9	2
10	3	10	2[b]	10	2
11	3	11	2.5	11	2
12	3.5	12	3	12	2.5
13	4.5[b]	13	---	13	3

a. Scale: 0 = Low background with few spikes to 4 = high background with many spikes.

b. Reject Bar

TABLE 15 (Cont'd.)

Qualitative Rating[a] of the Pulse Echo Charts for Lot M17

ID	Rating	ID	Rating	ID	Rating
1- 1	0	4- 1	0	7- 1	0
2	0.5	2	0	2	0.5
3	0.5	3	0.5	3	0.5
4	1	4	1	4	0.5
5	0	5	1.5	5	1
6	1.5	6	2	6	1.5
7	0.5	7	2	7	1.5
8	1	8	2	8	2
9	1	9	2	9	2
10	1.5	10	2.5	10	2
11	1.5	11	2.5	11	2.5
12	0.5	12	3	12	1.5
13	---	13	3	13	2
2- 1	0	5- 1	0	8- 1	0
2	0	2	0	2	0
3	0.5	3	1	3	0.5
4	0.5	4	1	4	1
5	0.5	5	1	5	1.5
6	1	6	1.5	6	2
7	1.5	7	2.5	7	2
8	2	8	2	8	2
9	1.5	9	2.5	9	2.5
10	1.5	10	2.5	10	2.5
11	2	11	3.5	11	3
12	2	12	3	12	3
13	---	13	---	13	4.5[b]
3- 1	0.5	6- 1	0	9- 1	0
2	0.5	2	1	2	0.5
3	0.5	3	0	3	1
4	1.5	4	1.5	4	1.5
5	1	5	1.5	5	1
6	1	6	2	6	2
7	2.5	7	2.5	7	2
8	2	8	2	8	1.5
9	2.5	9	2	9	1.5
10	2.5	10	2	10	1.5
11	2	11	1.5	11	1.5
12	1.5	12	2.5	12	2
13	---	13	---	13	---

a. Scale: 0 = Low background with spikes to 4 = high background with many spikes

b. Reject Bar

TABLE 15 (Cont'd.)

Qualitative Rating[a] of the Pulse Echo Charts for Lot M18

ID	Rating	ID	Rating	ID	Rating
1- 1	0	4- 1	1	7- 1	0
2	0.5	2	0.5	2	0.5
3	1	3	0.5	3	1
4	0.5	4	1	4	1
5	1	5	1.5	5	1.5
6	1	6	1.5	6	1
7	1.5	7	1.5	7	1.5
8	2	8	1.5	8	1.5
9	1.5	9	1	9	1.5
10	1.5	10	1.5	10	2
11	1.5	11	2	11	2
12	1.5	12	2	12	2
13	---	13	2.5[b]	13	---
2- 1	0	5- 1	0.5	8- 1	Billet
2	0.5	2	0.5	2	
3	0.5	3	0	3	8
4	1	4	1	4	
5	1	5	1	5	Installed
6	1.5	6	1	6	
7	2	7	1.5	7	in
8	2	8	1.5	8	
9	1.5	9	1.5	9	Extrusion
10	2	10	1.5	10	
11	2	11	1.5	11	
12	2	12	1	12	
13	---	13	4[b]	13	
3- 1	0.5	6- 1	0	9- 1	0
2	0	2	1.5	2	0.5
3	0.5	3	1	3	1
4	1	4	1	4	1
5	1	5	1.5	5	1
6	1	6	1.5	6	1
7	1	7	2	7	1.5
8	1.5	8	2	8	1.5
9	1	9	1.5	9	1.5
10	1.5	10	1.5	10	1
11	1.5	11	2	11	1
12	1	12	1	12	1.5
13	---	13	4.5[b]	13	1.5

a. Scale: 0 = Low background with few spikes to 4 = high background with many spikes.

b. Reject Bar

TABLE 15 (Cont'd.)

Qualitative Rating[a] of the Pulse
Echo Charts for Lot M19

ID	Rating	ID	Rating	ID	Rating
1- 1	0.5	4- 1	0	7- 1	0
2	1	2	1.5	2	0
3	1	3	2	3	1.5
4	1[b]	4	1	4	1.5
5	1.5	5	1.5	5	2
6	2	6	2	6	2
7	1.5	7	2	7	2
8	2.5	8	2	8	2
9	2.5	9	1.5	9	2.5
10	3	10	2.5	10	2.5
11	3	11	2.5	11	2
12	3	12	2.5	12	2
13	3	13	---	13	---
2- 1	0	5- 1	1.5	8- 1	0.5
2	0.5	2	1.5	2	0.5
3	0.5	3	1	3	1.5
4	1	4	1	4	1.5
5	2	5	2	5	2
6	2	6	1.5	6	2
7	1.5	7	2	7	2
8	2	8	2	8	2
9	2.5	9	2	9	2
10	2	10	2	10	3
11	2.5[b]	11	2.5	11	2
12	2	12	2.5	12	2
13	4.5[b]	13	---	13	---
3- 1	0	6- 1	0	9- 1	1.5
2	0	2	0.5	2	1
3	1.5	3	0.5	3	1
4	1	4	1.5	4	2.5[b]
5	1	5	1.5	5	2
6	1.5	6	1.5	6	2
7	1.5	7	2	7	2.5
8	2	8	2.5	8	3[b]
9	2.5	9	3	9	2.5
10	2.5	10	2	10	3
11	3	11	3	11	2.5
12	2.5	12	2	12	2[b]
13	---	13	---	13	---

a. Scale: 0 = Low background with few spikes to 4 ≈ high background with many spikes.

b. Reject Bar

TABLE 16

Average of Qualitative Ultrasonic Ratings

Billet	M15	M16	M17	M18	M19
1	1.38	1.54	0.75	1.13	1.96
2	1.21	1.69	1.08	1.33	1.77
3	1.54	2.12	1.46	0.96	1.58
4	1.58	1.33	1.69	1.38	1.75
5	1.12	1.77	1.71	1.27	1.79
6	1.08	1.67	1.54	1.58	1.67
7	1.77	1.79	1.35	1.29	1.67
8	1.67	1.13	1.88	----	1.75
9	1.04	1.65	1.33	1.08	2.13
All	1.38	1.64	1.43	1.26	1.79

TABLE 17

ECONOMIC ANALYSIS

CAPITAL EXPENDITURES:

Materials & Equipment			
Tank & Agitator		900	
Water & Oil Separator		50	
Hose		25	
Misc. Materials		50	
Ball Mill		12,000	
TOTAL MATERIALS & EQUIPMENT			$12,975
Labor			
Dept. 590	60 hrs. @ $30/hr. =	1,800	
Dept. 594	92 hrs. @ $30/hr. =	2,760	
Dept. 595	20 hrs. @ $30/hr. =	600	
TOTAL INSTALLATION & PROJECT LABOR			5,160
TOTAL CAPITAL OUTLAY			$18,135

OPERATION COSTS: [Per Melt]	Current	New	Change
Labor	*	*	*
Material	$21.38	$31.94	($10.56)
Yields	*	*	*
Per Melt (increase) in Direct Costs			($10.56)
Annual Basis			X500 melts/yr
ANNUAL IMPACT OF PROCESS CHANGE			(5,280)

* No impact or change due to different crucible coating process.

TABLE 18

Wash Performance*

Summary

Wash	Physical Properties	Firing Test	Reactivity Test	Cause for Rejection
		Zirconium Oxide ZrO_2		
Didier #1	F	--	--	Fast settling
Didier #2	F	--	--	Viscosity - thick
Didier #3	P	P	--	Resembled Remet #2
Remet #1	F	--	--	Viscosity - thick
Remet #2	P	P	(P)	Production wash
Remet #3	F	--	--	Viscosity - thick
ZYP "Z"	P	F	--	Firing tests
ZYP"ZO"	P	P	--	Resembled Remet #2
		Zirconium Silicate $ZrSiO_4$		
Foseco	P	P	--	Bacterial contamination; Si pickup
		Yttrium Oxide Y_2O_3		
"Rocky Flats"	P	P	--	Reference sample
45%-7M	P	F	--	Firing tests
45%-7M1	P	P	P	NMI chem/UT results
35%/50%-7M1	P	P	--	Resembled 45% Y_2O_3
45%-6CTL1	P	F	--	Firing tests
45%-DHT	F	--	--	Fast settling
45%-DM-LVT	B	F	--	Firing tests
ZYP "Y"	B	F	--	Firing tests
		Titanium Carbide TiC (Undercoat)		
ZYP	P	P	(P)	UT results
		Boron Nitride BN		
ZYP	P	F	--	Thermodynamically unstable

* Pass, Fail, Borderline

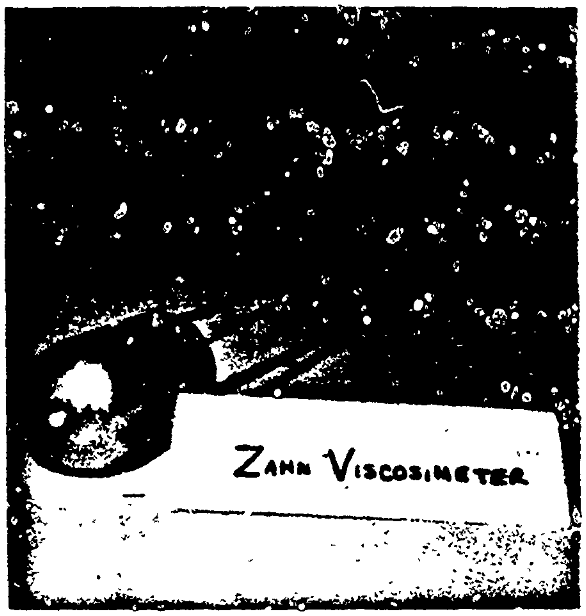


Figure 1. Zahn Viscosimeter

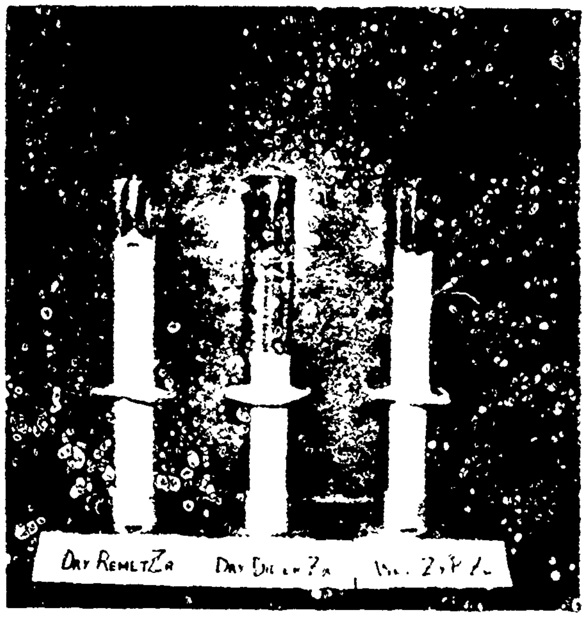


Figure 2. Settling Samples

Figure 3. Typical Painted Crucible

Figure 4. Results for a New Crucible
Fired at 1400°C

Figure 5. Results of Fired Crucible-30 Minutes Between Coats; Oven Dried

Figure 6. Results of Fired Crucible-Preheated; 30 Minutes Between Coats; Vacuum Dried

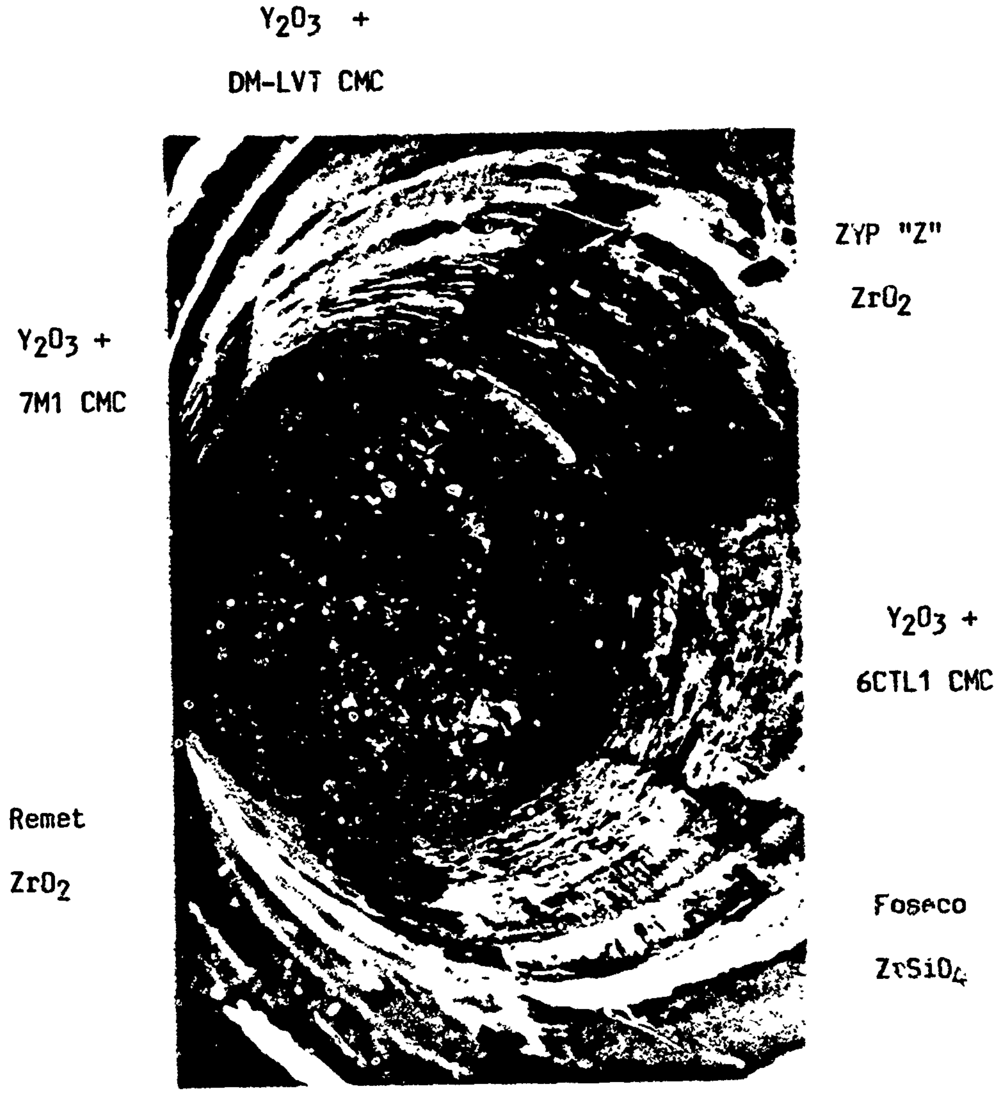
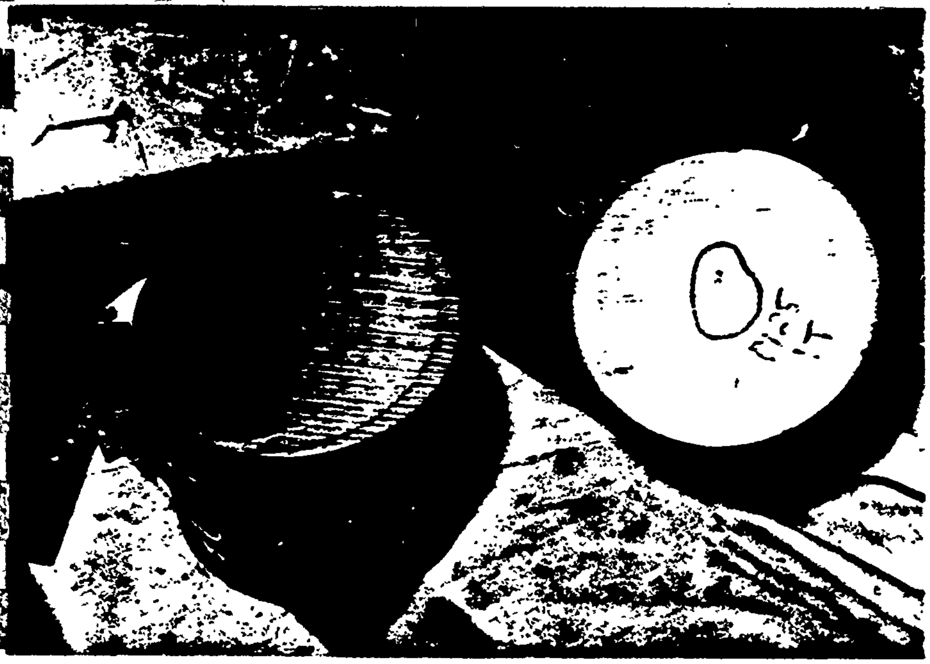


Figure 7. Results of Fired Crucible-Preheated; 20 Minutes Between Coats; Hood Dried

Figure 8. Results of Fired Crucible-Double Burnout; 20 Minutes Between Coats; Hood Dried



LOT N25

(Foseco $ZrSiO_4$)

Ingot 2 on left;

Ingot 1 on right

LOT N28

(Y_2O_3 + 7M1 CMC)

Ingot 2 in middle

Figure 9. Photographs Illustrating Ingot Porosity

LOT N28

(Y_2O_3 + 7M1 CMC)

Ingot 2 on top;

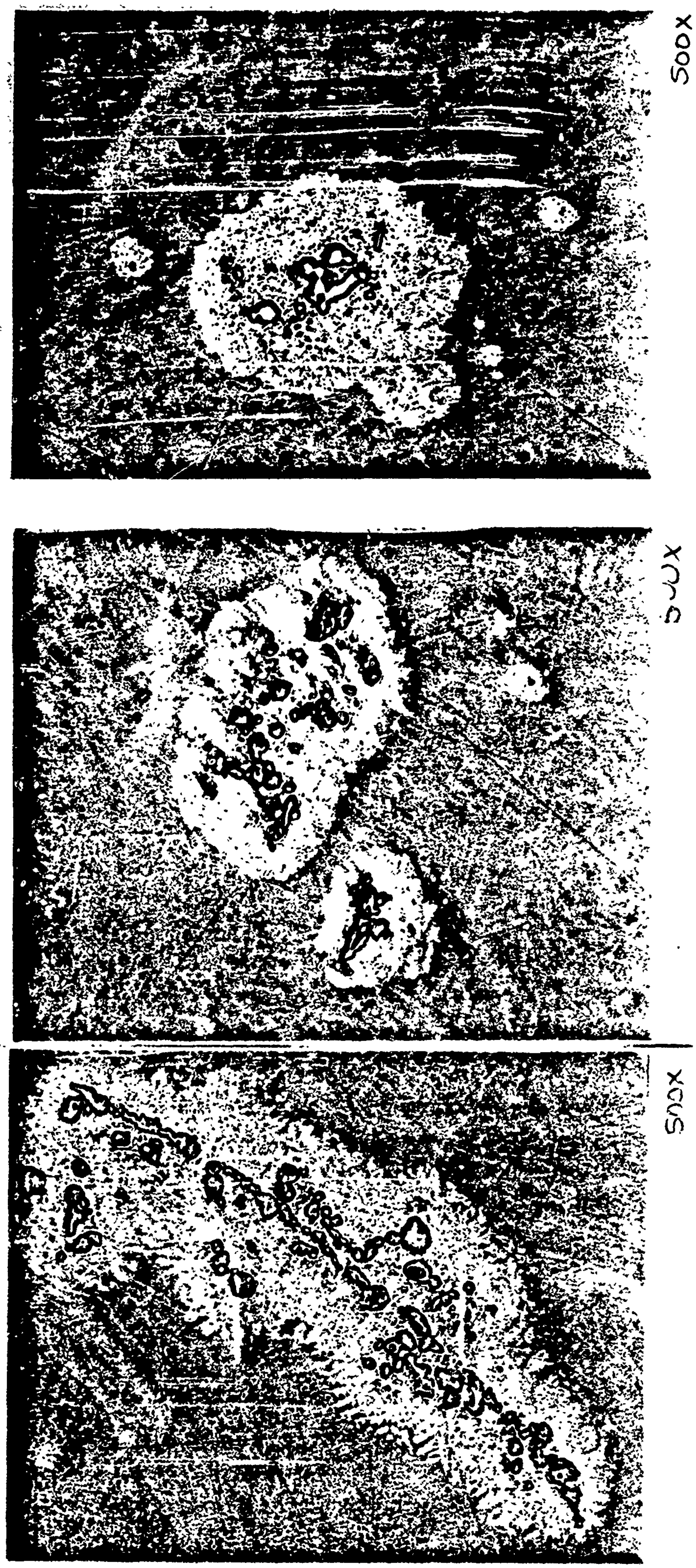
Ingot 1 on bottom

LOT N28

(Y_2O_3 + 7M1 CMC)

Ingot 2

Figure 10. Photographs Illustrating Ingot Porosity

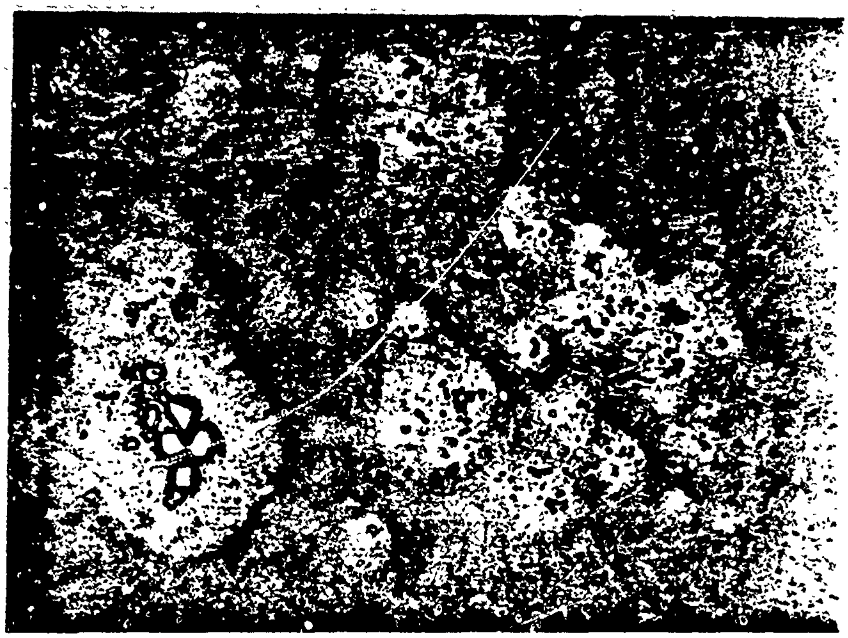


Edge

Mid-Radius

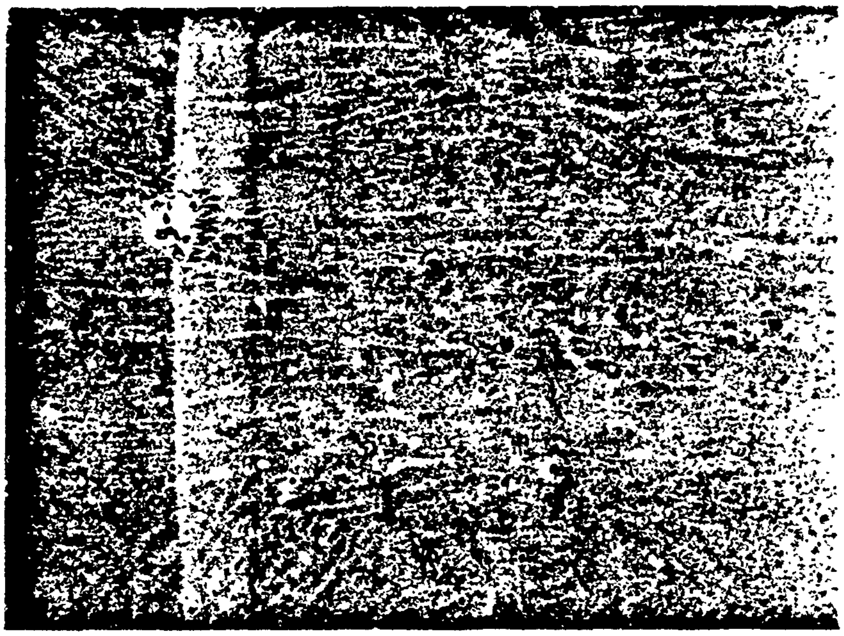
Center

Figure 11.
Photomicrographs of
an N74 Ingot Top



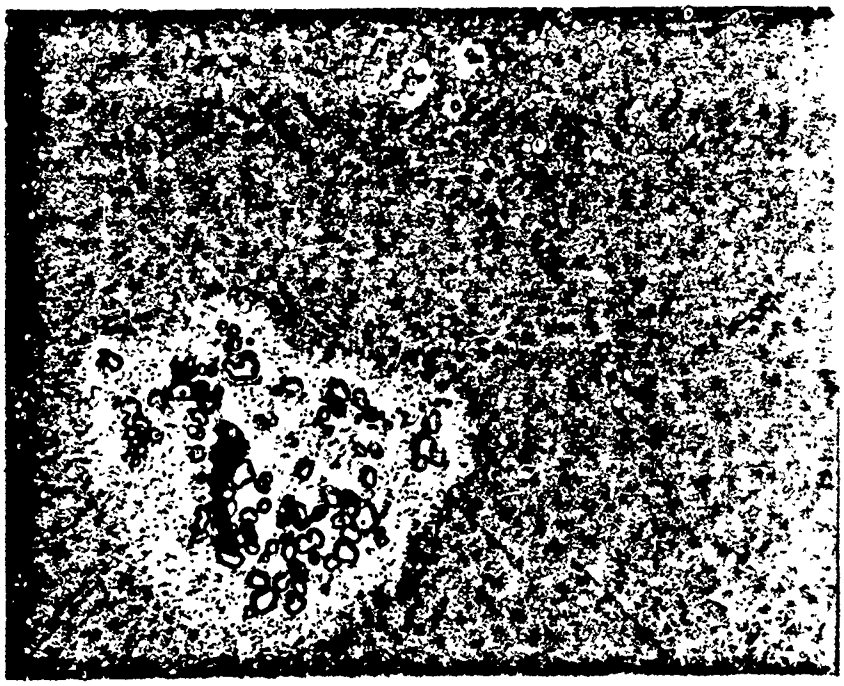
500X

Edge



500X

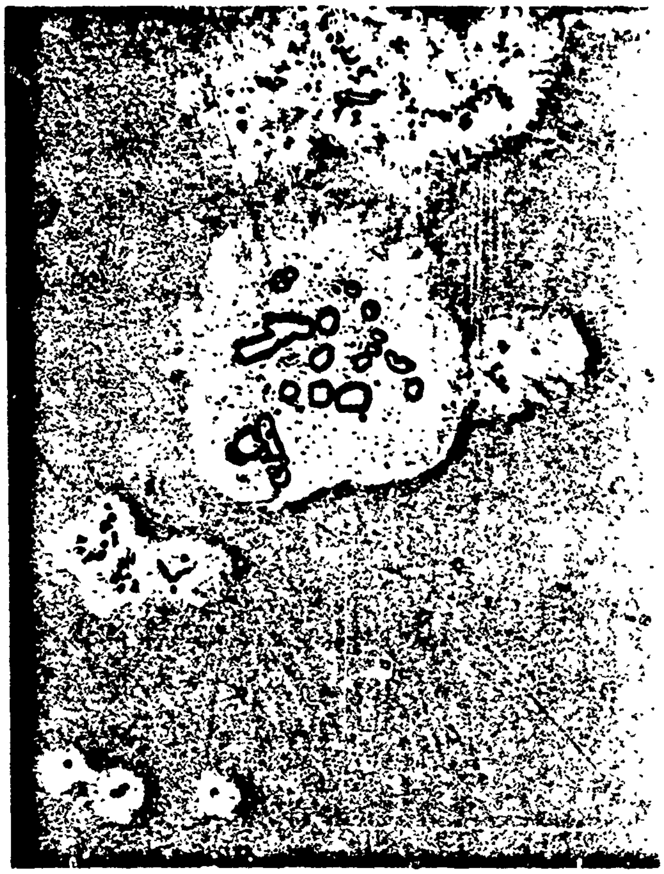
Mid-Radius



500X

Center

Figure 12.
Photomicrographs of
an N77 Ingot Top



600X

Figure 13.

Photomicrographs of an N77 Ingot Bottom

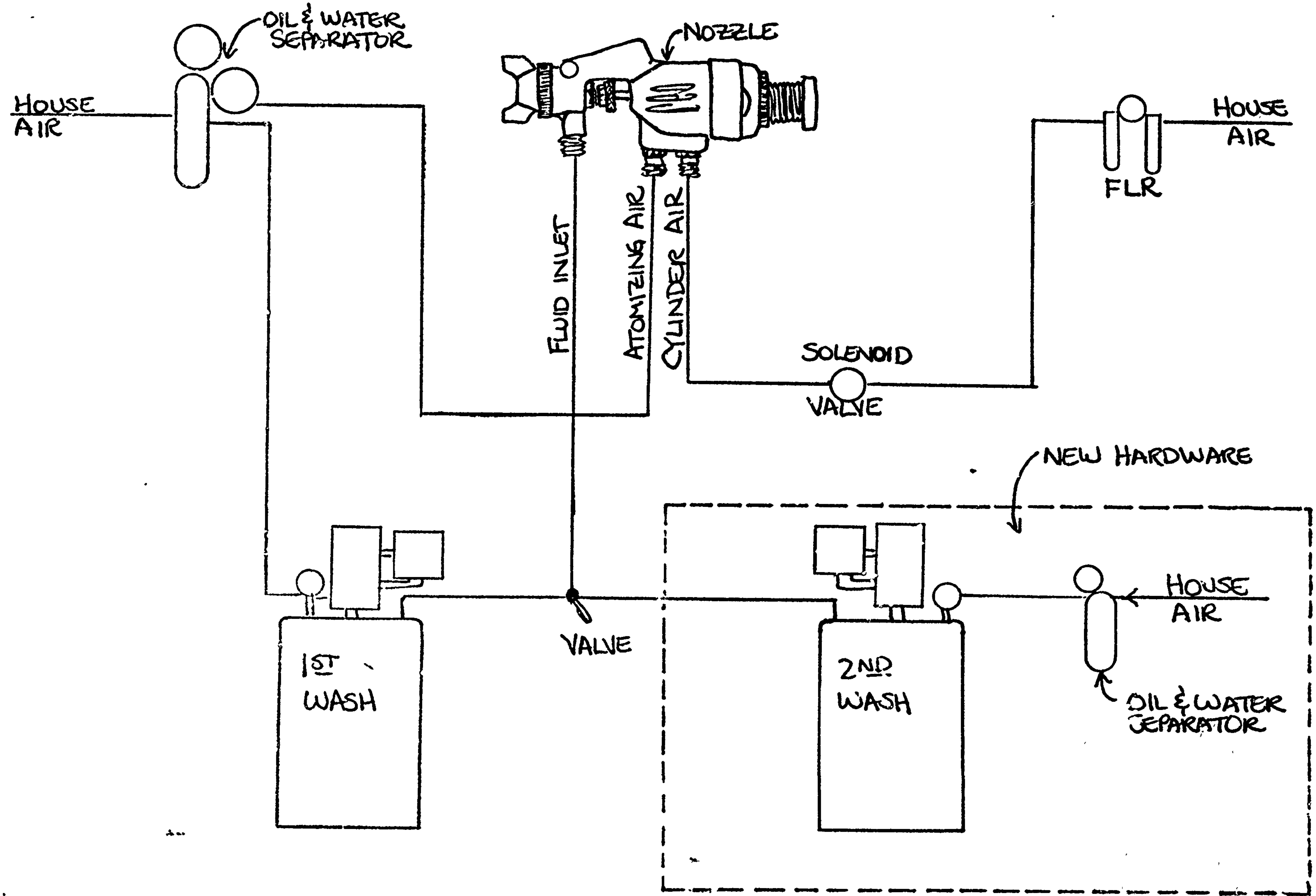


Figure 14. Schematic of Crucible Coating Station Depicting Conversion to a Two Wash System

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